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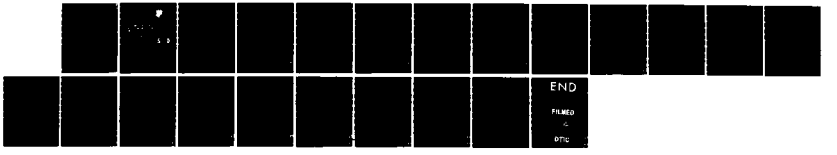

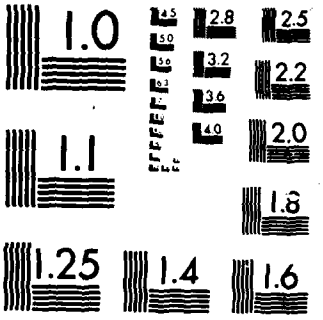

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RADC-TR-85-238
In-House Report
November 1985

THE EFFECTS OF ARSINE PRESSURE ON THE COMPOSITIONS, CARRIER CONCENTRATIONS, MOBILITIES AND GROWTH RATES OF EPITAXIAL LAYERS OF $Ga_xIn_{1-x}As$ PREPARED BY THE VPE-HYDRIDE TECHNIQUE

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19. ABSTRACT (Continue on reverse if necessary and identify by block number)

The effect of three arsine partial pressures on the preparation of the ternary, $Ga_xIn_{1-x}As$, was investigated. Operational parameters are given for the preparation of $Ga_{0.47}In_{0.53}As$ at these pressures. Increased arsine partial pressures decreased the gallium composition of the ternary. Growth rates have been determined as a function of gallium monochloride and arsine partial pressures. Increased mole fractions of arsine do not lower carrier concentrations or increase mobilities of the $Ga_xIn_{1-x}As$ layers.

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11. TITLE (Contd)

Mobilities, and Growth Rates of Epitaxial Layers of $Ga_xIn_{1-x}As$ Prepared by the VPE-Hydride Technique

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The Effects of Arsine Pressure on the Compositions, Carrier Concentrations, Mobilities and Growth Rates of Epitaxial Layers of $Ga_xIn_{1-x}As$ Prepared by the VPE-Hydride Technique

1. INTRODUCTION

Active epitaxial layers of $Ga_xIn_{1-x}As$ have many physical and electronic characteristics that warrant their use in optoelectronic and micro- and millimeter-wave devices. Photodetectors with $Ga_{0.47}In_{0.53}As$ layers have the ability to detect wavelengths of light where silica fibers exhibit their lowest losses.[1,2] Multiple-quantum well lasers with $Ga_{0.47}In_{0.53}As$/InP structures have exhibited low threshold current requirements and low temperature sensitivity.[3,4] High speed field-effect transistors with $Ga_xIn_{1-x}As$ layers prepared for application in

(Received for publication 18 November 1985)

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the micro- and millimeter-wave region have been reported.[5-7] Future developments will undoubtedly find greater uses of $Ga_xIn_{1-x}As$ epitaxial layers in semiconductor devices.

$Ga_xIn_{1-x}As$ epitaxial layers can be grown by a number of techniques, e.g., LPE,[8] MBE,[9] MOCVD,[10] and VPE.[11,12] The VPE technique can either use the group V trichloride (chloride method) or the group V hydride (hydride method) as the source of the group V element. In the hydride method, hydrogen chloride is used to convert the group III elements to their chlorides. The chloride technique has been shown to give higher quality products, but Abrokwah and co-workers[13] have recently demonstrated that the hydride is also capable of producing quality layers. The hydride technique has the added advantage that the sources of the group V elements are gaseous and can easily be controlled.

The hydride technique has been studied extensively since its inception in 1966 by Tietjen and Amick.[14] Recently, questions have arisen concerning the preparation of the ternary, $Ga_xIn_{1-x}As$, by the hydride technique: (1) Is there a mole fraction effect in the preparation of $Ga_xIn_{1-x}As$?, and (2) What is the effect of

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AsH_3 pressures on the compositions of the ternaries? The primary purpose of the study was to investigate these areas. In addition, the study reports the operational parameters for the preparation of the $Ga_{0.47}In_{0.53}As$ ternary and includes growth rates of the ternaries at three different arsenic pressures.

DiLorenzo and Moore[15] originally observed that increased arsenic mole fractions decreased the carrier concentrations and increased the mobilities of GaAs epitaxial layers. The III-V semiconductors were prepared by the vapor-phase epitaxy-chloride method. These studies were interpreted to show that the major background dopant was silicon. Kennedy, Potter, and Davies[16] later demonstrated the mole fraction effect in the preparation of GaAs by the hydride method. This effect was further confirmed in the hydride process by Pogge and Kemlage.[17] These authors show that the impurities decreased not only by increased pressures of the arsenic species, but also by hydrogen chloride. The magnitude of the doping level changes with hydrogen chloride were found to be smaller. Pogge and Kemlage attributed the lower impurity level with higher arsenic pressures to a blocking effect of the arsenic species in preventing impurity incorporation. The mole fraction effect has also been observed with InP layers.[18] No data is available to demonstrate the mole fraction effect in the preparation of $Ga_xIn_{1-x}As$ ternaries. Preliminary results reported in this paper on the preparation of $Ga_xIn_{1-x}As$ indicate that the mobility (or carrier concentration) is not affected by the mole fraction of the arsenic vapor.

S. B. Hyder[19] reported that the indium-hydrogen chloride must be increased to maintain the lattice-matched $Ga_{0.47}In_{0.53}As$ when increased arsenic pressures are used. Thermodynamic and experimental studies[20-22] have shown that the

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indium concentration in $Ga_xIn_{1-x}As$ increases with AsH_3 pressures. The present study, with similar operational parameters as those of Hyder,[19] indicates that the indium mole fraction in the ternary does increase with AsH_3 pressure.

2. EXPERIMENTAL

The epitaxial layers of $Ga_xIn_{1-x}As$ on InP substrates were prepared with a double-barrel quartz reactor. The quartz reactor, shown in Figure 1 along with its temperature profile, is basically a three zone reactor: source, mixing, and deposition. The zones are heated by a series of seven tubular-type resistance heating units. Each unit consists of two Lindberg half-circle heating elements. The temperature of the heating units is controlled by a system consisting of seven Eurotherm temperature controllers (model 919) that trigger seven silicon controlled rectifiers (model 931). The temperature controllers receive inputs from Omega chromel-alumel thermocouples (scass-020-60) placed at the mid-points

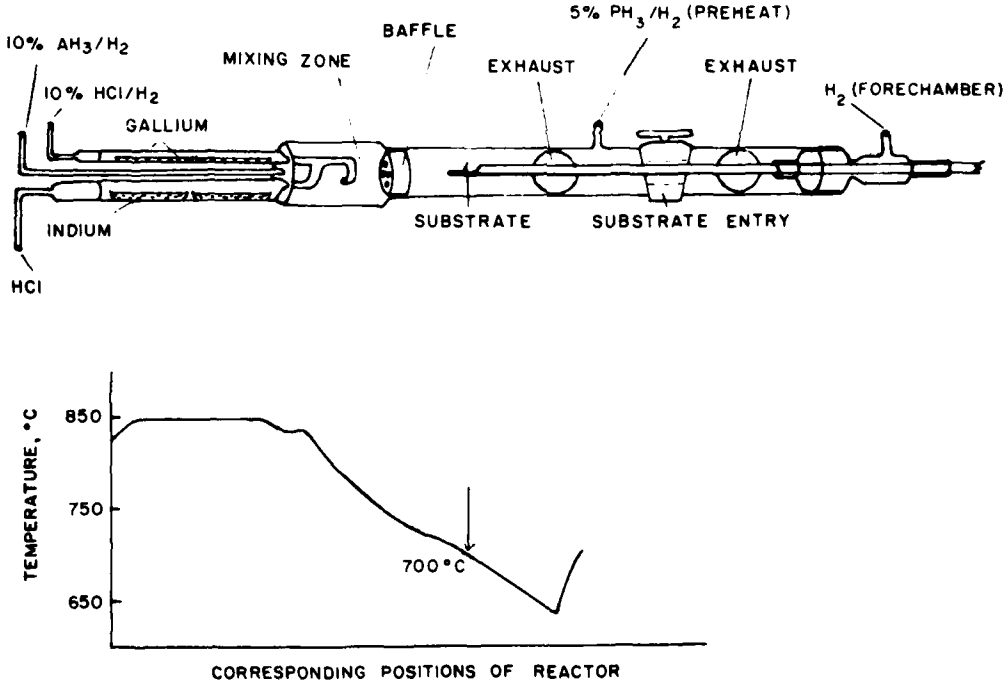

Figure 1. Double-Barrel Reactor (Upper Figure) With Corresponding Temperature Profile (Lower Figure)

of the heating units near the quartz reactor. The temperatures used throughout the study are shown in the lower portion of Figure 1. The source zone was maintained at 850°C, while the mixing zone varied from 850°C at the source zone end to 725°C at the start of the deposition zone. The substrates were held at 700°C in the deposition zone.

The reactant gas flows were regulated by a custom-built gas control unit made by Niagara Scientific Company, Syracuse, N.Y. The gas control system contains 14 Tylan mass flow controllers (FC-260) and a Tylan Tymer 16 Programmable Sequencer that is able to program the 14 mass flow controllers through 96 time intervals. The 14 mass flow controllers give the system the versatility to prepare quaternary compounds of the III-V elements with n and p doping. Only nine of the mass flow controllers were used in the present study. All the reactant gases used were of the highest purity obtainable. The carrier gas, hydrogen (99.9999 percent), was further purified by a hydrogen purifier (Palladium Diffusion Process - Englehard). Indium (99.9999 percent) in the quartz boat was converted to its monochloride by flowing a mixture of 6.3 sccm of 100 percent HCl (99.999 percent) in 200 ccm hydrogen over the metal surface. The flow rates of 10 percent HCl/H_2 over the gallium metal (99.9999 percent) varied from 5.4 ccm to 11.4 ccm with an additional 200 ccm of hydrogen. A mixture of 10 percent AsH_3/H_2 with hydrogen was added to the mixing zone. The flow rates of 10 percent arsine/hydrogen investigated were 25, 75, and 225 ccm. The volume of hydrogen was adjusted to keep the total volume of gases relatively constant (1440 ± 3.5 ccm). Prior to deposition, 100 ccm of 5 percent PH_3/H_2 with 50 ccm of hydrogen was added to the preheat zone in order to inhibit the decomposition of the InP substrate when in the preheat zone. Olsen and Zamerowski[23] and Yamauchi and Susa[24] showed that the surface morphology of InP substrate can be maintained at higher temperatures if the substrate is in a PH_3 atmosphere.

The substrates were obtained from Liquid Encapsulated Czochralski (LEC) grown iron-doped InP boules. Slices of the boules were cut 2-3° off the (100) plane towards the (110) plane. The preparation of the substrates prior to growth experiments is described in Ref. 25.

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The procedure used to grow $Ga_xIn_{1-x}As$ epitaxial layers was begun by placing the substrate slightly beyond the substrate entry port and equilibrating the reactor with hydrogen. After 10 min, the growth mixture was initiated with the addition of 300 ccm hydrogen at the forechamber, and 100 ccm of 5 percent PH_3/H_2 plus 50 ccm hydrogen at the preheat zone. The substrate was placed in the preheat zone at a temperature of 620°C for 28 min. The PH_3/H_2 flow was terminated, and, after one min, the substrate was placed in the deposition zone. The reaction time was usually 30 min, but the duration of some of the runs was 10 and 20 min.

The composition of the epitaxial layers was determined from the lattice parameters using Vegard's Law. The lattice constants were determined by x-ray diffractometry with CuK α irradiation, using InP as an internal standard. An EDAX study of the cross-section of a cleaved sample indicated some phosphorus displacement during processing.

Growth rates were determined from the surface of the substrate, mass of deposit, density of the ternary, and time of growth. Consideration of the formation of epitaxial layers on both sides of the substrate was taken into account in the calculation of growth rates.

Carrier concentrations (n) and mobilities (μ) were determined at room temperature from the resistivity and Hall measurements with the van der Pauw technique.[26] A permanent magnet provided a field of 4 kG for the Hall measurements.

3. RESULTS AND DISCUSSION

Various operational parameters were evaluated initially in order to obtain quality epitaxial layers. The parameters used in this study are similar to those of Hyder, et al.[27] Figure 2 shows the composition of the various ternaries formed as a function of gallium monochloride partial pressure at three different As_4 partial pressures. The concentrations of the monochlorides were calculated using the equilibrium constants derived from the thermodynamic data reported by Kirwan[28] and Seki et al.[29] The values for As_4 are 1/4 of the pressure of AsH_3.

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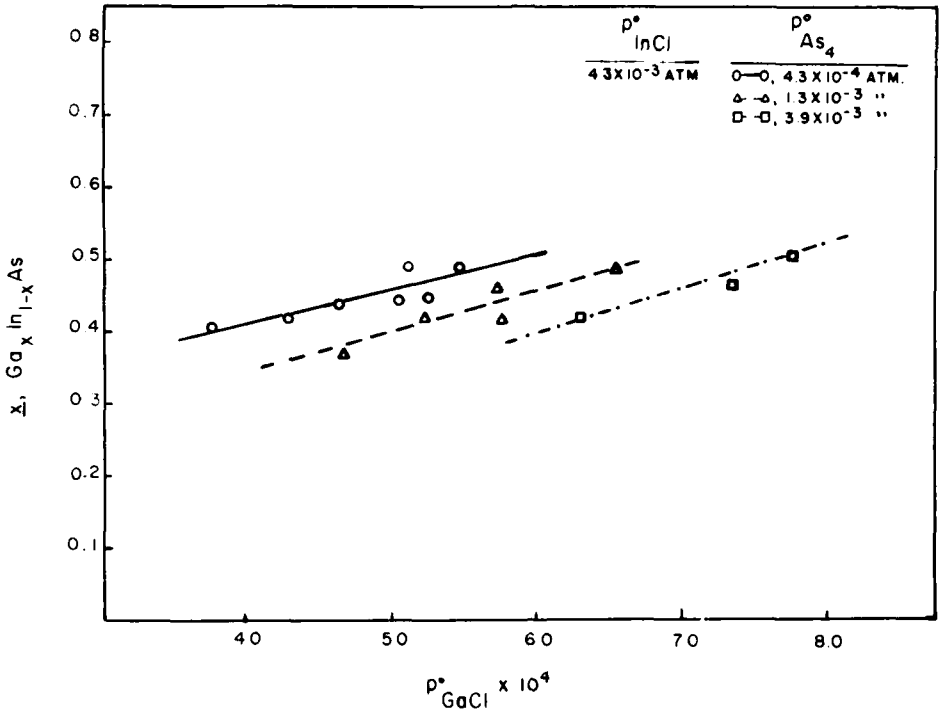

Figure 2. Composition of Ternaries, $Ga_xIn_{1-x}As$, as a Function of Gallium Chloride Pressures at Three Different As_4 Partial Pressures. Indium chloride pressure, 4.1×10^{-3} atm. Total gas flow, 1440 ± 3 ccm. Source zone temperature, 850°C; mixing zone temperature, average 750°C; and deposition zone temperature, 700°C. Reaction time, 10, 20, or 30 min

At As_4 constant pressures, an increase of GaCl partial pressure by 1.5 times increases the gallium mole fraction in $Ga_xIn_{1-x}As$ from approximately 0.4 to 0.5 in a linear manner. The data in Figure 2 show that increased As_4 pressure decreases the mole fraction of gallium in the ternary. The parameters for the growth of $Ga_{0.47}In_{0.53}As$ on InP substrates at the three arsenic pressures are given in Table 1. Hyder et al[27] found that, at a P°_{InCl} of 4.8×10^{-3} atm and $P^\circ_{As_4}$ of 4.6×10^{-4} atm, a P°_{GaCl} of 4.8×10^{-4} atm was needed for the preparation of $Ga_{0.47}In_{0.53}As$. This value is in agreement with the present study, where P°_{GaCl} was 5.3×10^{-4} atm and P°_{InCl} and $P^\circ_{As_4}$ were 4.6×10^{-3} and 4.3×10^{-4} atm, respectively.

Hyder et al[27] observed that increasing the $P^\circ_{As_4}$ required an increased pressure of indium monochloride to maintain the $Ga_{0.47}In_{0.53}As$ composition. Our data (Figure 2) indicate that an increase in gallium monochloride pressure would be necessary to maintain the ternary composition. The experimental

Table 1. Growth Parameters for the Preparation of $Ga_{0.47}In_{0.53}As$ on InP (Fe-Doped) Substrate at Constant $P°_{InCl}$ (4.3×10^{-3} atm). Source temperature, 850°C; mixing temperature, avg 750°C; and deposition temperature, 700°C. Total gas flow rate, 1440 ± 3 ccm

$P°_{InCl}$ (atm)	$P°_{GaAs}$ (atm)	$P°_{As_4}$ (atm)
4.3×10^{-3}	5.3×10^{-4}	4.3×10^{-4}
4.3×10^{-3}	6.3×10^{-4}	1.3×10^{-3}
4.3×10^{-3}	7.3×10^{-4}	3.9×10^{-3}

results of Nagai,[21] as well as their thermodynamic modelling calculations, showed an increase in indium in the ternary with increased As_4 partial pressures. This result is further supported by the modelling calculations of Kajiyama[22] and Jones and Tu.[30] The reason for the discrepancy between the observations of Hyder et al,[27] ours, and others[21,22] is not known.

The growth rates of the $Ga_xIn_{1-x}As$ layers are depicted in Figure 3. The data show that increasing the GaCl pressure decreases the growth rate of the ternary layer at the lower two As_4 pressures. As the As_4 pressure is increased, the rate of decrease of the growth rate is decreased. The growth rate at $P°_{As_4} = 3.9 \times 10^{-3}$ atm is relatively constant with increasing gallium monochloride pressures. Shaw[31] has demonstrated that increasing the gallium monochloride partial pressure in the kinetically-limited region decreases the deposition rate of the formation of GaAs. Shaw attributed this decrease in GaAs growth to a competitive process where GaCl molecules are absorbed at the arsenic receptor sites, thereby hindering growth. Figure 3(c) gives substance to this model: at $P°_{GaCl}$ values greater than 6.4×10^{-4} atm, the Langmuir-type absorption of GaCl probably reaches saturation. Under these conditions, the growth depends on the dissociation of GaCl from the arsenic active site. The number of reactive sites at these partial pressures remains relatively constant, resulting in a slowly decreasing growth rate with increasing GaCl pressures. Nagai's modelling[21] suggested that the deposition rate should increase with As_4 pressures; this could not be verified in the present study, since the GaCl pressure appears to be the predominant factor affecting the growth rate.

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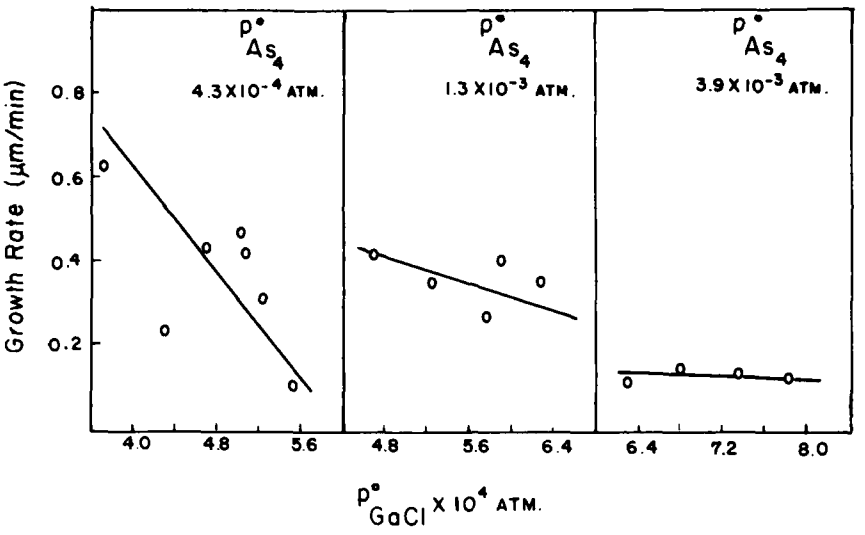

Figure 3. Growth Rates for the Formation of the Ternaries, $Ga_xIn_{1-x}As$ as a Function of Gallium Chloride Partial Pressure at Three As_4 Partial Pressures

The effects of As_4 pressure on the carrier concentrations and mobilities of the various $Ga_xIn_{1-x}As$ ternaries are shown in Figures 4 and 5. Figure 4 shows that increasing the $P^{\circ}As_4$ from 4.3×10^{-3} atm to 1.3×10^{-3} atm increases the room temperature carrier concentrations of the different ternaries. Since these experiments were carried out at relatively the same partial pressures of GaCl, the only factor influencing the carrier concentrations is the arsenic mole fraction. These results imply that the mole fraction effect of arsine is not playing a role in the preparation of $Ga_xIn_{1-x}As$. A possible reason for the increase in carrier concentrations with increasing As_4 pressures is the presence of n-type impurities in the arsine gas.

Figure 5 depicts the mobilities of the ternaries, $Ga_xIn_{1-x}As$, as a function of composition. The plot illustrates that increasing the As_4 partial pressure has no effect on the mobilities of the epitaxial layers. This was not unexpected, since the carrier concentrations exhibited an absence of a mole fraction effect. Figure 5 is reminiscent of the plot reported by Whiteley and Ghandhi,[32] where the maximum mobilities are observed when the indium mole fraction is greater than 0.53.

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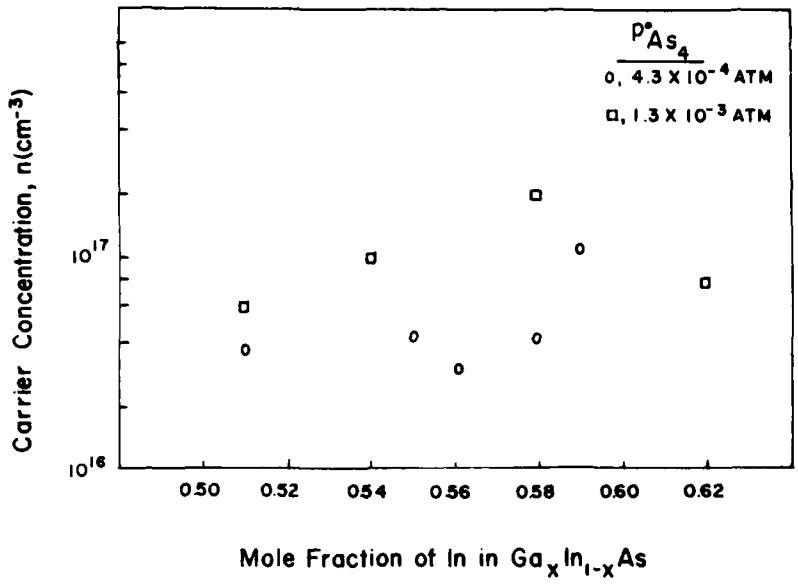

Figure 4. Carrier Concentrations (Room Temperature) of Ternaries, $Ga_xIn_{1-x}As$, as a Function of Composition. Preparation parameters in Figure 2

These higher mobilities have been attributed to the observation that less dislocation formation occurs in layers under compressive stress.[33] This same effect was also reported by Erstfeld et al.[12] The mobilities of the ternaries determined at 77K increased , on an average, approximately 100 $cm^2/v\text{-}sec$. These low mobilities at 77K are probably the result of excess scattering by the high concentration of ions and neutral impurity atoms. Calavanov and Siukaev[34] showed that scattering by ion and neutral impurities plays an important role at low temperatures. The layers prepared at $P^{\circ}_{As_4}$ of 3.9×10^{-3} atm showed carrier concentrations in the mid $10^{16} cm^{-3}$, but exhibited lower mobilities than those reported in Figure 5. This behavior can possibly be attributed to high compensation ratios.

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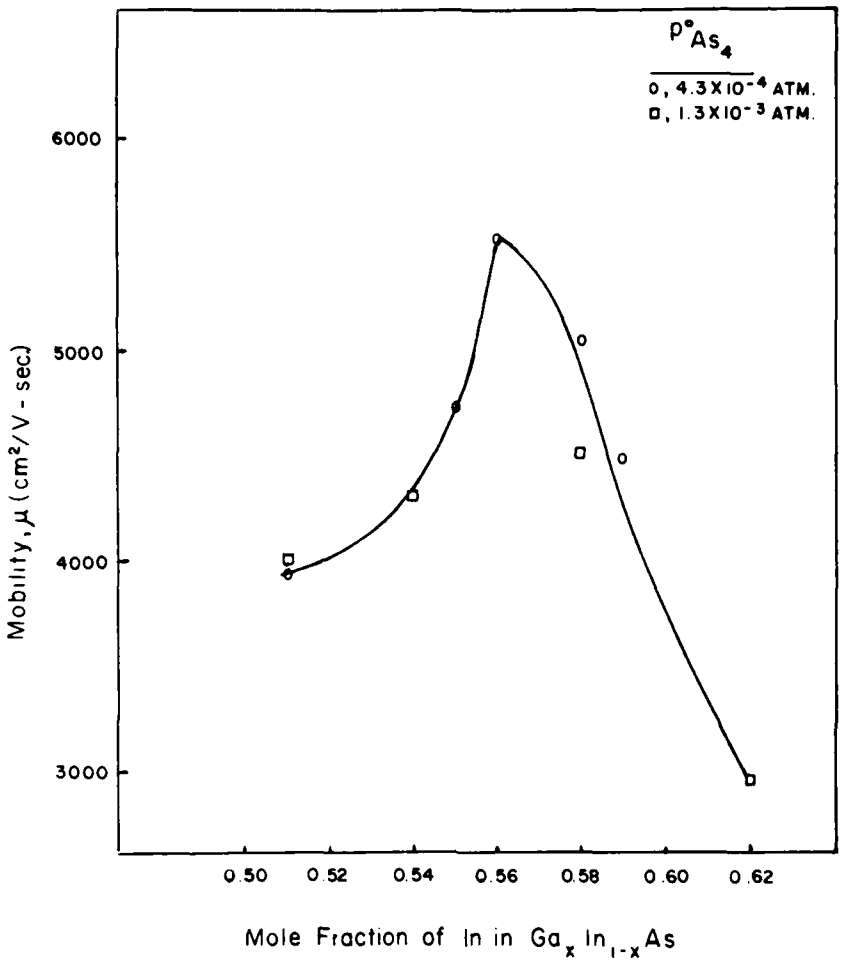

Figure 5. Mobilities (Room Temperature) of Ternaries, $Ga_xIn_{1-x}As$, as a Function of Composition. Preparation parameters in Figure 2

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